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#### **Patent**

#### **CLAIMS**:

What is claimed is:

- 1. A method for regulating the concentration of water in the acid feed stream in a process for the production of HF by the reaction with a fluorine containing material comprising:
  - (a) providing an acid feed stream comprising acid, bound water and free water;
- (b) estimating the concentration of bound water and free water in the acid feed stream by providing a probe in at least a portion of said feed stream; and
- (c) adjusting the concentration of free water in said acid feed stream based on said estimating step.
- 2. The method of claim 1 wherein said probe interacts with said portion of said acid feed stream and generates an information signal based on said interaction.
- 3. The method of claim 2 wherein said information signal is functionally related to the concentration of water in the acid feed stream and wherein said estimating step comprises electronically computing an estimated concentration of water in said acid feed stream on the basis of the interaction between said probe and said acid feed stream.
- 4. The method of claim 1 wherein said probe comprises a diamond-tipped ATR probe.

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- 5. The method of claim 1 wherein said acid in said feed stream comprises sulfuric acid, hydrofluoric acid, and fluorosulfonic acid.
  - 6. The method of claim 5 wherein said acid feed stream is a liquid phase stream.
- 7. The method of claim 1 wherein said adjusting step (c) comprises introducing a drying agent into said acid feed stream.
  - 8. The method of clam 7 wherein said drying agent comprises oleum.
- 9. The method of claim 8 wherein said introducing step comprises introducing an amount of oleum sufficient to convert substantially all of said free water in said stream to sulfuric acid.
- 10. The method of claim 1 wherein said adjusting step (c) comprises introducing additional water into said acid feed stream.
- 11. A method for controlling water content of an acid comprising the steps of:
  - (a) providing an acid;
- (b) determining the concentration of water in said acid using an in-line measurement system; and

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- (c) adjusting the concentration of water in said acid based on the concentration of water measured in step (b).
- 12. The method of claim 11 wherein said acid comprises sulfuric acid, hydrofluoric acid, fluorosulfonic acid, and water.
  - 13. The method of claim 11 wherein said in-line measurement system comprises a computer and an infrared analyzer equipped with a diamond-tipped probe.
  - 14. The method of claim 11 wherein said adjusting step (c) comprises adding a drying agent to said acid mixture to form a dried acid mixture comprising a concentration of free water lower than the concentration determined in step (b).
    - 15. A method for manufacturing hydrofluoric acid comprising the steps of:
      - (a) providing an acid mixture;
  - (b) determining the concentration of free water in said acid mixture using an in-line measurement system;
  - (c) adding a drying agent to said acid mixture to form a dried acid mixture comprising a concentration of free water lower than the concentration determined in step (b);
  - (d) reacting said dried acid mixture with fluorspar to form hydrofluoric acid.

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- 16. The method of claim 15 wherein said acid mixture comprises sulfuric acid, hydrofluoric acid, fluorosulfonic acid, and water.
- 17. The method of claim 15 wherein said in-line measurement system comprises a computer and an infrared analyzer equipped with a diamond-tipped probe.
  - 18. A method for manufacturing hydrofluoric acid comprising the steps of:
  - (a) providing a stream comprising sulfuric acid, hydrofluoric acid, fluorosulfonic acid and water;
  - (b) determining the concentrations of free water and bound water in said stream using an in-line measurement system comprising a computer and an infrared analyzer equipped with a diamond-tipped ATR probe;
    - (c) adjusting the concentration of free water in said acid feed stream based on said estimating step.
  - 19. The method of claim 18 wherein said adjusting step comprises introducing an amount of sulfur trioxide sufficient to convert substantially all of said free water in said stream to sulfuric acid.